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FRICTIONAL PROPERTIES OF SYNTHETIC MOS AND A NEW LUBRICATING COMPOUND WS 2

by

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EDITED TRANSLATION

FRICTIONAL PROPERTIES OF SYNTHETIC MOS AND A NEW LUBRICATING COMPUND WS

By: Zdzislaw Has

Translated by: Contract F33657-68-D-0865

TT8001099

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further studies which will establish its superiority over other lubricating compounds. As concerns machine parts or mechanisms of such type that greases or oils cannot be used, it appears that the MoS, or WS, lubricating compounds are the only ones which can be used. Prior to use of synthetic WS, in actual practice a number of tests should still be conducted to round out the data about its chemical properties; in particular, the temperature at which oxidation in air begins should be accurately determined. Initial studies for the determination of the temperature at which oxidation begins show that it falls between 400 and 450°C, and therefore that the temperature is of the same order as the oxidation temperature of molybdenum disulphide. As a special application of lubricating compounds of this type we should mention all indicating instrumentation and other devices where the quantity of heat generated during operation is small, that is, where the task of the lubricating oils is not to carry heat away but solely to lutricate the parts. The degree of lubricating properties of natural molybdenum disulphide cited in the literature of western nations should be looked at critically, since in this case industrial advertisement plays an important role. However, in certain cases deskribed in the introduction and conclusion of this article, it appears that the MoSo and WoS, compounds could be used successfully in our industry. The use of these compounds as additives to lubricating oils requires further studies.

FRICTIONAL PROPERTIES OF SYNTHETIC MoS₂ AND A NEW LUBRICATING COMPOUND WS₂

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This paper discusses the studies of the friction coefficient for natural and synthetic molybdenum disulphide (NoS₂) as well as for a new lubricating compound and namely tungsten disulphide (WS₂). Studies conducted by the author did not exhibit any differences in the friction coefficients of the above three compounds.

Molybdenum disulphide was introduced as a lubricating agent to American industry in 1950 by A. Sonntag [1] under the name "Nolykote." Since then the use of MoS₂ in American industry has been increasing continuously. It has also aroused interest in Europe, in particular in England and Germany. First studies, in Poland, on the application of molybdenum disulphide to outting fluids were published by A. Latour and R. Nadej [8]. Later, the works of Prof. N. Halaunbrenner and his coworkers were published; this department is engaged in the study of lubricating properties of MoS₂ [8, 4].

LUBRICATING PROPERTIES OF NATURAL MOLYBDENUM DISULPHIDE AND ITS INDUSTRIAL APPLICATIONS

Since the physicochemical properties of molybdenum disulphide, as well as its lubricating properties, have already been discussed in our writings [2, 3 and 4] we will cite in the remainder of this paper only those studies on the application of MoS2 which have not yet been published. In this category we find mainly the studies of G. Spengler [5] who carried out a relatively large number of diverse studies on the applications of MoS2. Figure 1 shows a graph characterizing the coefficient of friction μ as a function of the sliding velocity under a constant pressure of 8858 kg/cm2 (which was calculated according to the Hertz equation) for the case of a molybdenum disulphide layer, a graphite layer, a clean steel surface and a surface lubricated with oleic acid [5]. Both molybdenum disulphide and graphite were applied to the steel disk in the form of a paste consisting of an MoS2 or graphite powder dissolved in an appropriate substance which evaporates at higher temperatures. After application of a layer of the paste, the disks were heated at a temperature of 350°C; then, after the disks cooled off, the excess was scraped off, leaving a thin MoS2 or graphite layer.

At present we are seeking methods of bonding the molybdenum disulphide to steel surfaces as strongly as possible. Among other things, tests carried out on impregnation, with molybdenum disulphide, of surfaces previously phosphated [6, 7] resulted in significant reduction of the friction sliding coefficient. For instance, with pressures of 0.5 kg/cm² the friction sliding coefficient was 0.10, whereas with pressures of 19 kg/cm² it was 0.04.

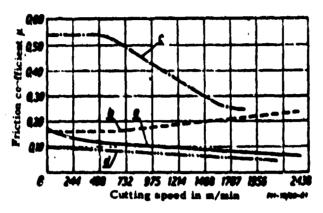


Fig. 1. Coefficient of friction μ as function of sliding speed under a load of 8858 kg/cm² calculated with the Hertz equation [5]: a) an MoS₂ layer; b) a graphite layer; c) dry steel; d) steel covered with oleic acid CH₂(CH₂)₇ • CH:CH(CH₂)₇ COOH.

Other noteworthy studies [8] are those conducted with various lubricating compounds and conducted by forcing a 1.001" diameter steel pin (hardened, polished and honed) into a sleeve of 1.000" diameter. The results of these studies are tabulated in Fig. 2. These studies enabled us to determine the coefficients of friction as a function of lubricant used, of the force required to insert the pin, as well as of lubricant behavior.

The shaded areas in the graph indicate that the contact surfaces were scratched or marred. Press fitting was done on a hydraulic test stand. The insertion speed was 1.5 cm/min and in all cases it was a fixed quantity. As can be seen from the figure, molybdenum disulphide is definitely the best of the lubricating compounds used in these tests. Mixing MoS₂ with heavy oils or greases is not advantageous since it interferes in the bonding of molybdenum disulphide crystals with the friction surfaces.

Studies on sintering with MoS₂ additives also yielded interesting results [8]. Powdered MoS₂ was mixed in various ratios with silver and copper powders (Cu, 5%; MoS₂, from 0 to 35%; and Ag, from 60 to 95%); then it was sintered at a temperature of 760°C and at a pressure of 470 kg/cm². Studies of the friction coefficients of these sintered materials, conducted under various sliding speeds and loadings, show that the friction coefficient decreases as the MoS₂ content increases. For instance, the friction coefficient of a sintering which contained no MoS₂ was μ = 0.30; with an MoS₂ content of 3%, μ = 0.22, and with an MoS₂ content in the sintering of 35%, μ = 0.16. It should be mentioned that starting with an MoS₂ content of 5% the friction coefficient was already essentially independent of sliding speed and loading

pressure.

Incorporation of molybdenum disulphide into synthetic materials allows us to lower synthatially their coefficients of friction [9]. For instance, addition of small quantities of MoS₂ to methyl polymetacrylate reduces its friction coefficient from 0.45 to 0.1-0.15; furthermore, with sliding of the pure substance, the surface is damaged in a very short time and remains marred. On the other hand, the presence of MoS₂ causes a thin film to be formed on the surface of the substance, so that even after a long period if time the friction coefficient does not exceed 0.15. Similarly, small MoS₂ additions to ebonites (in such quantities as not to reduce their mechanical properties) improve their riction coefficient from 0.40 to 0.25.

Molybdenum disulphide has been tested industrially as a lubricating agent. Threaded connections operating at higher temperatures and lubricated with molybdenum disulphide require lower forces for tightening, and furthermore the threads remain clean and unmarred. According to a number of publications, MoS₂ prevents seizing of threaded connections operating even at 600°C [8].

Molybdenum disulphide was also used successfully in cold drawing of materials and in difficult pull broaching to lubricate threads carrying high loads, threaded connections, cones and wedges, center holes and connecting elements in fitted and moving machine parts, as well as for sliding parts in precision machining.

Commercially, molybdenum disulphide is obtained in the form of a mixture containing 70% finely powdered MoS2 and 25% light mineral oil with a stabilizer additive. The base material can also consist of ethylene glycol, which easily evaporates upon heating, and which burns out without any residues at higher temperatures. This mixture finds application for practically all metals and synthetics except natural and synthetic rubber, leather and lacquer. It is characterized by very good lubricating properties at low and medium temperatures, under air flow conditions, under rubber seals on shafts, etc. This paste also has anticorrosive properties [8].

In liquid suspension, molybdenum disulphide can be used in an oil can to grease moving oven parts.

Molybdenum disulphide, having a large dispersion (grain size from 1 to 3 μ), was used successfully as an additive for combustion engine fuels.

The addition of two per cent MoS₂ to greases used for heavy geared transmission results in very good performance [8]. For instance, heavy engaging gears lubricated with a grease without the MoS₂ additive heated up so much after three hours of operation that the meshing characteristics were altered to such an extent that gear shavings were produced or interference was present. On the other hand, use of a 2% MoS₂ additive permitted continuous operation of the transmission for 9 hours without overheating.

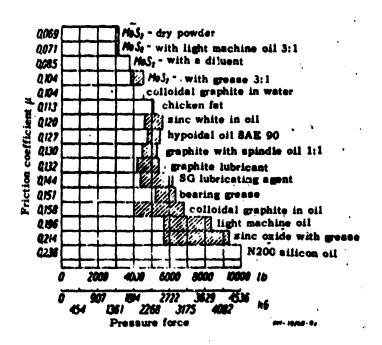


Fig. 2. Friction coefficient as a function of force magnitude required to press a pin into a sleeve when using various lubricating agents. The shaded portions on the graph indicate the occurrence of scratching on the contact surfaces [8].

The following possibilities for further application of MoS₂ should be cited:

- connection of parts with narrow tolerance limits;
- lubrication of matrices for synthetic resins, lubrication when the lubricant should be electrically nonconducting;
- lubrication of moving parts in vacuum where liquid or graphite lubricants are inapplicable.

ASSUMPTIONS IN PROPERTY STUDIES

Extensive studies on the lubricating properties of natural molybdenum disulphide (a purified ore product) already conducted, and its increasing application in the industry of technologically advanced countries such as the United States, England and Germany, induce one to devote more attention to this problem.

One reason for undertaking this work involved the proposals voted on at a science and technology conference on the subject of "cutting fluids and cooling methods in machine cutting" which in the realm of research work suggests the necessity of investigating the use of molybdenum disulphide in lubricating liquids [10]. Direct results of this work were theoretical predictions showing that other chemical compounds could exhibit lubricating characteristics similar to those of MoS₂ if only they had a crystalline structure similar to that of MoS₂.

The conclusions of some investigators that synthetic molyb-

denum disulphide does not possess any lubricating properties because of its abnormal crystalline structure [8] also acted as an inducement to resolve this problem. In this case it was necessary only to produce synthetic molybdenum disulphide having the normal crystalline structure, since then its lubricating properties should be the same as those of natural molybdenum disulphide.

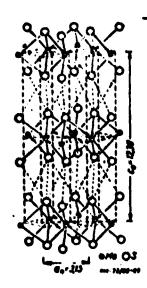


Fig. 3. Crystallographic structures of MoS2, WS2, MoSe2 and WSe [11, 12, 15].

Among all existing compounds whose crystallographic structures are known, there are only four which have a structure identical to that of molybdenum disulphide, i.e., they crystallize in a nexagonal pattern with a C7 net type and D_{6h} symmetry that is C6/mmc (Fig. 3). These compounds are: MoS₂, WS₂, MoSe₂ and WSe₂ [11, 12].

Of all these compounds, the one most widely studied, for obvious reasons, is molybdenum disulphide (MoS₂), known as molybdenite (MoS₂).

MoS, hardness is 1 to 1.5 on the Moss scale, its specific weight is 4.7 to 4.8 g//cm³ and it cleaves easily. It is insoluble in both cold and hot water as well as in mineral oils and synthetic lubricating agents. It is difficul to dissolve in acids, and is soluble only in aqua regia, hydrochloric acid, chlorine and fluorine. In pure oxygen it oxidizes easily at room temperature; however, in air it oxidizes only at temperatures from 400°C up. The melting point of molybdenum disulphide is 1185°C. In vacuum at a temperature of 1100°C it disintegrates into metallic molybdenum and

free sulphur. In a rare gas atmosphere (e.g., argon) the melting point of MoS₂ is 1427°C. Molybdenite is electrically nonconductive and nonmagnetic [5].

Tungsten disulphide is a grayish-black powder which when crushed forms a steel gray film with metallic luster. WS2 crystals are easily spread and stick easily together, they are soft and leave a dirty streak. This compound forms a strongly bonded layer on glass. When heated in the presence of air, WS: changes very slightly. When heated to very high temperatures in an electric oven it slowly loses its sulphur. In vacuum at a temperature of 1100°C it still does not decompose, but at a temperature of 1200°C it loses 60% sulphur in a matter of 2 hours, and at a temperature of 2000°C it sheds its sulphur very fast. When heated in air, at first it becomes gray, and then under intense heating, it forms green WO: In a hydrogen atmosphere, reduction starts only at a temperature of 800°C and H2S is formed in the process. Tungsten disulphide is resistant to the action of gaseous HCl, of hydrochloric acid, of hydrogen fluoride and nitric acid; it is insoluble in water [13].

Molybdenum diselenide (MoSe₂) does not dissolve in hot hydrochloric acid. In hot concentrated sulphuric acid it disintegrates causing evolution of SO₂. It is insoluble in ammonia, al-

kali and sulphides. It dissolves alightly when boiled in concentrated alkali. Oxidizing agents attack MoSe₂ much less than MoSe₃. Molybdenum dioxide does not exhibit any superconductivity to a temperature of 78°K [13].

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Fig. 4. X-ray photographs of compounds discussed in the article "Mikrometa," Cu Ka; 40 kv; 14 ma; 40 min: a) synthetic MoS₂ obtained according to literature procedures; b) natural MoS₂ from the paste "Molykote Paste G"; c) synthetic MoS₂ obtained according to the technological procedure developed by the author; d) synthetic WS₂ obtained according to the technological procedure developed by the author.

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Fig. 5. View of instrumentation used to study the sliding coefficient of friction, together with suspended frame.

Tungsten dioxide (WSe₂) has not been studied much and the only information pertaining to its chemical properties that can be found in open literature is that its crystall lattice is of the same type as that of MoS₂. The source literature (Lieb. Ann. 116 [1860] 122) could not be found in our libraries [13].

From among the above cited compounds it has been decided to investigate primarily the lubricating properties of tungsten disulphide, since at present its physicochemical properties are better known than those of MoSe; and WSe;. Furthermore, the manufacture of selenium compounds is more costly than the manufacture of

sulphur compounds, if only because of the higher cost of selenium. For this reason also, their eventual application in practice seems improbable.

It appears that the study of the lubricating properties of MoSe; and WSe; will be only of purely academic interest because, on the basis of theoretical considerations, it does not appear as if these compounds could have better lubricating properties than sulphur compounds.

STUDIES OF PROPERTIES

1. Obtaining Molybdenum Disulphide ${\rm MoS}_2$ and Tungsten Disulphide ${\rm WS}_2$

In the production of synthetic compounds MoS_2 and WS_2 we used chemically pure raw materials. In making molybdenum disulphide we used ammonium molybdate (NH₄)₆ Mo_7O_{24} ·6H₂O whereas for WS_2 we used ammonium tungstate.



Fig. 6. Schematic diagram of the instrumentation used to study the coefficient of friction.

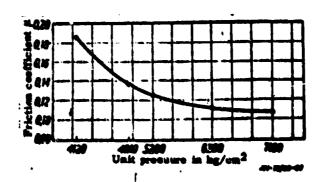


Fig. 7. Dependence of the coefficient of friction μ of natural molybdenum disulphide on contact pressure for a constant sliding speed ν = 482.6 m/min.

In both cases, to obtain MoS₂ and WS₂ we could use the metal oxides MoO₃ and WO₂; however, since they are difficult to obtain, we used ammonium compounds. The methods cited in the literature [13, 14] for obtaining molybdenum disulphide coincide. However, the MoS₂ obtained in this way has an irregular crystallographic structure (Fig. 4a) which differs greatly from the crystallographic structure of natural MoS₂ (Fig. 4b). This fact clarifies

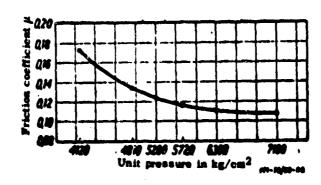


Fig. 8. Dependence of the coefficient of friction μ of synthetic molybdenum disulphide on contact pressure for a constant sliding speed ν = 482.6 m/min.

the findings of some investigators [8] that synthetic molybdenum disulphide has no lubricating properties. In reality, this compound (Fig. 4a) does possess lubricating properties but these are very inferior to those of molybdenite (natural MoS₂) which can be easily verified by rubbing it between the fingers.

It was necessary therefore to develop a technology for obtaining MoS₂ such that its crystallographic structure be regular, i.e., it should not differ from the crystallographic structure of mineral molybdenite.

Figure 4c shows an x-ray photograph of molybdenum disulphide obtained by the techniques developed by the author. As can be seen by comparison of Figs. 4b and 4c, the crystallographic structures of both compounds are identical.

Figure 4d shows the x-ray photograph of tungsten disulphide obtained synthetical? by the author. From a comparison of the x-ray photograph of Figs. 4b, 4c and 4d it can be assumed that the lubricating properties of these compounds are very close. The x-ray photographs were taken with an x-ray machine of the Mikromet type, by the Debye-Scherer method, using a tube with a copper anticathode. The exposure conditions were as follows: 40 kv, 14 ma, 40 min.

A powder of natural molybdenum disulphide was obtained by extraction from the base substance of the Molykote Paste, G-Molykote Produktions GmbH München.

2. Measurement of the Friction Coefficient

The measurement of the sliding friction coefficient was performed on the device shown in Fig. 5; its schematic diagram is shown in Fig. 6. The measurement method is based on the harmonic vibration of a slat on top of two wheels of identical diameter and separated by a distance of 21. The wheels are set into motion at the same angular speed but in opposite direction; as a result of this, a sliding frictional force arises between the rotating wheels and the slat. In the case of unsymmetrical slat position (that is, if the center of gravity of the slat is displaced by a

distance x from the central position) the force F_A < F_B and this gives rise to slat motion.

Since the slat has some inertia it will move further away than dictated by the distribution of forces F_A and F_B . This gives rise to return motion of the slat, and therefore to generation of vibrations. Considering the position of the slat as shown in Fig. 6 and assuming that its weight is G kg, we can write:

$$R_A = G \cdot \frac{l-s}{2l} kG$$

$$R_B = G \cdot \frac{l+s}{2l} kG$$

$$F_A = \mu \cdot G \cdot \frac{l-s}{2l} kG$$

$$F_B = \mu \cdot G \cdot \frac{l+s}{2l} kG$$

where μ is the friction coefficient being sought.

From Newton's second law we get that $F_A + F_B = m \cdot a_e$ where m is the mass of the slat and a_e is the acceleration of its center of gravity.

Summing the components along the horisontal axis we get:

$$P_A - P_B = m \cdot \frac{\sigma_B}{4\sigma}$$

Substituting the values of F_A and F_{B^*} we get:

$$p \cdot 6 \frac{1-a}{2l} - p \cdot 6 \frac{1+a}{2l} = \frac{6}{a} \cdot \frac{6a}{46}$$

from which:

Fig. 9. Dependence of the coefficient of friction μ of synthetic tungsten disulphide on contact pressure for a constant sliding speed $\nu = 482.6$ m/min.

Setting $\mu g/l = k^2$, we get:

The general solution of this equation is as follows:

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Constants A and B can be determined from the initial conditions; $A = \sqrt{\frac{EE}{i}}$ is the periodic frequency of the vibrations by means of which we can express the period of the oscillations as $T = 2\pi/k$.

Hence
$$T = 2\pi \cdot \sqrt{\frac{1}{\mu \cdot s}} \cdot \exp = \frac{4\pi^2 \cdot l}{sT^2} \sim 4.02 \, l/T^2$$

The diameters of the wheels used in the device are identical and equal to 98 mm. The separation of the axes of the two wheels is 125 mm. The rotational speeds of the two wheels were also the same and equal to 1555 rpm. Therefore, the relative sliding velocities were 482.6 m/min.

In the studies of the friction coefficient we used a steel rod 4 mm in diameter, mounted in a frame, as shown in Fig. 5, instead of the slat. Then the unit contact force was computed with the Hertz equation:

$$\sigma_{max} = \alpha \sqrt{\frac{P \cdot E^2}{R_1^2}}$$

and with a frame load of from 1320 g to 6680 g, the contact force varied between the limits of 4120 kg/cm^2 and 7100 kg/cm^2 .

Suspensions of natural or synthetic powders of NoS2 or synthetic WS2 in toluene were applied with a brush to the well polished and degreased surfaces of the wheels. After evaporation of the toluene the remaining powder on the wheels was rubbed in with a gla : rod. After such a treatment, the wheel surfaces had a metal c gray luster.

The frame period of oscillation was measured with a stopwatch accurate to 0.1 sec. Three measurements were taken for each loading, and the average value was used in the computation of the friction coefficient shown on the graphs, and in each case the oscillati period was computed from the time for ten cycles. Differences in time for the experiments conducted in this way varied between the limits of 0.1 and 0.3 sec for the ten cycles.

The graphs (in Figs. 7, 8 and 9) show the coefficients of friction for the three lubricating agents mentioned as a plot of friction coefficient versus unit load for a constant sliding speed v = 482.6 m/min.

As can be seen from these drawings all three curves have identical shape and do not differ from each other significantly

which proves that the lubricating properties of a compound are influenced mainly by its crystallographic structure. The fristion coefficient in the case of steel wheels and a steel rod, untreated with any lubricating agent, is 0.54.

Figure 10 shows the surface of the wheel where the layer of synthetic molybdenum disulphide is destroyed. This destruction occurred after 20 min operation of the device to measure the coefficient of friction, and it manifested itself by a pronounced change in the period of frame oscillation. Figure 11 shows, similarly to Fig. 10, a destroyed layer of tungsten disulphide after 25 min of oscillation. Therefore it can be concluded on the basis of the cited photographs (Figs. 10 and 11) and from the time to failure of the lubricating layer, be it molybdenum disulphide or tungsten disulphide, that even with such a primitive method of impregnation of steel surfaces very good results are obtained.

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Fig. 10. View of destroyed lubricating layer of synthetic molybdenum disulphide after 20 min of sliding under a loading of 7100 kg/cm 2 ; magnification $60\times$.

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Fig. 11. View of destroyed lubricating layer of synthetic tungsten disulphide after 25 min of sliding under a loading of 7100 kg/cm 2 ; magnification $60\times$.

A practical method of testing the lubricating properties of molybdenum disulphide and tungsten disulphide obtained by the author was to use these compounds to grease the metal-rubber bushings in the spring suspension of both front and rear wheels of a Skoda-1102 automobile. Since the metal-rubber bushings cannot be lubricated with any greasy lubricants, the unlubricated parts have damaged both the metal and rubber bushings after only 3000 km of driving. After application of either the molybdenum disulphide or tungsten disulphide as lubricating agent, neither the metal nor rubber bushings showed any traces of wear after 6000 km.

CONCLUSIONS

It may be concluded on the basis of the tests conducted that the governing factor in the lubricating properties of solid compounds is their crystallographic structure; this was verified by studies of the friction coefficient for molybdenum disulphide and tungsten disulphide.

These tests also prove that synthetically obtained MoS₂ and WS₂ compounds can be used as lubricating agents just as well as natural MoS₂ (molybdenite).

Studies of the properties of WS2 and MoS2 also contribute to the solution of the problem of surface sulphiding of steel. In many scientific studies dealing with the problem of sulphiding steels it has been noted that tools made of high-speed steels were more wear resistant when subjected to surface sulphiding. Since high-speed steels contain a large percentage of tungsten (9% to 18%) it is very probable that during the sulphiding process, besides forming iron and manganese sulphides, tungsten disulphide is also formed and its lubricating properties are responsible for decreased wear on the tools.

Increased application of MoS₂, be it synthetic or natural, in our industry must be based on further studies which will establish its superiority over other lubricating compounds. As concerns machine parts or mechanisms of such type that greases or oils cannot be used, it appears that the MoS₂ or WS₂ lubricating compounds are the only ones which can be used.

Prior to use of synthetic WS: in actual practice a number of tests should still be conducted to round out the data about its chemical properties; in particular, the temperature at which oxidation in air begins should be accurately determined. Initial studies for the determination of the temperature at which oxidation begins show that it falls between 400 and 450°C, and therefore that the temperature is of the same order as the oxidation temperature of molybdenum disulphide.

As a special application of lubricating compounds of this type we should mention all indicating instrumentation and other devices where the quantity of heat generated during operation is small, that is, where the task of the lubricating oils is not to carry heat away but solely to lubricate the parts.

The degree of lubricating properties of natural molybdenum

disulphide cited in the literature of western nations should be looked at critically, since in this case industrial advertisement plays an important role. However, in certain cases described in the introduction and conclusion of this article, it appears that the MoS₂ and WoS compounds could be used successfully in our industry. The use of these compounds as additives to lubricating oils requires further studies.

This work was performed at the Metallurgy Department of the Polytechnic of Lodz. The author wishes to thank the head of the department, Prof. Z. Wendorffow, for a number of valuable suggestions and advice during this work.

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